Docket No: 33343-01
Patent

Assistant Commissioner for Patents
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Washington, D.C. 20231

New Application for Transmittal

Transmitted herewith for filing is the patent application of the following Inventor(s): Guanglin Sun, Fakhruddin Ahmed and Bruce Christian Black ; For: Improved Coated Pesitidal Matrices, A Process for Their Preparation and Compositions Containing Them.

1. Papers enclosed which are required for a filing date under 35 CFR 1.53(b):
   - 43 Pages of specification
   - ☐ Sequence Listing (__ pages)
   - 7 Pages of claims
   - 1 Page(s) of abstract
   - Sheets of drawing
     - ☐ Formal
     - ☐ Informal

2. Additional papers enclosed
   - ☒ Information Disclosure Statement
   - ☒ Form PTO-1449
   - ☒ Citations
   - ☐ Declaration of Biological Deposit
   - ☐ Computer Readable Form of Sequence Listing
   - ☐ Declaration Under 37 CFR 1.821(f)
   - ☐ Other:

3. Declaration
   - ☒ Enclosed and executed by all inventor(s) (in two counterparts)
   - ☐ Not enclosed or not executed by all inventor(s)

CERTIFICATION UNDER 37 CFR 1.10

I hereby certify that this paper and the documents referred to as enclosed therein are being deposited with the United States Postal Service on the date written below in an envelope as "Express Mail Post Office to Addressee" Mailing Label Number EM165270628US addressed to the Assistant Commissioner for Patents, Box Patent Application, Washington, D.C. 20231.

6-9-98
Date

Barbara L. Renda

4. Assignment
An assignment of the invention to:

AMERICAN CYANAMID COMPANY
Five Giralda Farms
Madison, New Jersey 07940-0874

☐ was made in the prior application and recorded in PTO on , Reel , Frame .

☐ is attached under separate Recordation Form Cover Sheet.

☒ will follow.

5. Filing Fee Calculation

CLAIMS AS FILED				
(1) FOR	(2) NUMBER FILED	(3) NUMBER EXTRA X RATE		(4) BASIC FEE
				$790.00
TOTAL CLAIMS	35	15	X $ 22.00	330.00
INDEPENDENT CLAIMS	2	0	X $ 82.00	0
MULTIPLE DEPENDENCY FEE			X $ 270.00	0
			Total Filing Fee:	$1120.00

6. Method of Payment of Fees:
Charge Deposit Account No. 01-1300 in the amount of $1120.00
A duplicate of this transmittal is attached.

7. Instructions as to Overpayment:
Credit any overpayment to Deposit Account No. 01-1300.

8. General Authorization:
During the pendency of this application treat any reply requiring a petition for extension of time for its timely submission as containing a request therefor for the appropriate length of time. The Commissioner is hereby authorized to charge all required extension of time fees during the entire pendency of this application to Deposit Account No. 01-1300.

9. Authorization to Charge Additional Fees

☒ The Commissioner is hereby authorized to charge the following additional fees by this paper and during the entire pendency of this application to Deposit Account No. 01-1300:

☒ 37 CFR 1.16(a), (f), or (g) filing fees

☒ 37 CFR 1.16(b), (c), and (d) presentation of extra claims

☒ 37 CFR 1.16(e) surcharge for filing the basic filing fee and/or declaration on a date later than the filing date of the application.

☒ 37 CFR 1.17 application processing fees

10. Relate back (35 USC 119(e))

☒ Amend the Specification by inserting before the first line the sentence:

--This application claims priority from copending provisional application(s) serial number 60/052,071 filed on July 9, 1997.--

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33343-01

IMPROVED COATED PESTICIDAL MATRICES, A PROCESS FOR THEIR PREPARATION AND COMPOSITIONS CONTAINING THEM

BACKGROUND OF THE INVENTION

Certain pesticidal agents are inactivated by ultraviolet radiation from the sun. Because those pesticidal agents are useful for the control of pests and are applied in areas where they will be exposed to ultraviolet radiation, there is a need for photostable compositions containing those agents.

To prevent ultraviolet inactivation of pesticidal agents, compositions have been prepared which contain ultraviolet absorbers and/or reflectors and a pesticidal agent.

U.S. Patent 3,541,203 describes a protected virus composition for insect control. The preferred composition includes a virus, an actinic light absorbing material and a polymeric binder material. However, the process used to prepare the preferred compositions of U.S. Patent 3,541,203 requires the use of toxic materials and numerous washing steps with flammable solvents thus making it unsuitable for commercial manufacture.

U.S. Patent 4,948,586 discloses a microencapsulated insecticidal pathogen. Four microencapsulated compositions are shown to decrease the photoinactivation of *Autographa californica* NPV. However, the microencap-

sulated compositions retain only from 30.7 to 71.43% of the original activity upon exposure to sunlight. U.S. Patent 4,948,586 discloses a method of preparing microencapsulated insecticidal pathogens which has numerous steps and is both time-consuming and laborious. It is apparent that neither the process, nor the microencapsulated insecticidal pathogens, described in U.S. Patent 4,948,586, are entirely satisfactory for providing a product stable to ultraviolet radiation.

U.S. Patent 5,560,909 discloses a process for the preparation of insecticidal compositions which requires the modification of the charge of a charged polymer to precipitate the polymer and entrap the insecticide. However, this process is not entirely satisfactory because a small amount of the functional groups on the polymer will remain charged in the final product, resulting in a less efficacious product.

EP 697170-A1 discloses a process for the preparation of coated pesticidal agents which requires that the coating polymer be completely dissolved and which adjusts the pH of the coating solution to attain such dissolution. Unfortunately, such dissolution reduces some of the desirable properties of the coating polymer, resulting in a less efficacious product.

SUMMARY OF THE INVENTION

The present invention comprises an improved process for the preparation of a coated pesticidal matrix, which process comprises: a) preparing an aqueous mixture comprising a pesticidal agent, a pH-dependent polymer and water, wherein the pH is below the solubilization pH of the polymer; and b) drying the aqueous mixture to produce the coated pesticidal matrix. The aqueous mixture optionally includes a plasticizer, an ultraviolet protector, an activity enhancer and/or a glidant thus

resulting in their presence in the coated pesticidal matrix. Preferably, the pesticidal agent is a particulate chemical insecticide or a viral, bacterial or fungal insecticidal pathogen.

The present invention also comprises wettable powder pesticidal compositions which comprise coated pesticidal matrices, together with suitable carriers.

The present invention further comprises a method for improving the residual control of a pest comprising the application of a matrix made by the process of this invention.

It is an object of the present invention to provide a coated pesticidal matrix which retains the desirable properties of the coating polymer and thus retains a significant amount of its original pesticidal activity after exposure to ultra-violet radiation.

It is also an object of the present invention to provide an improved process for the preparation of a coated pesticidal matrix under mild conditions which avoid degradation of the pesticidal agent.

Other objects of this invention will be apparent to those skilled in the art from the following description and the appended claims.

DETAILED DESCRIPTION OF THE INVENTION

The improved process of this invention comprises:

a) preparing an aqueous mixture comprising a pesticidal agent, a pH-dependent polymer, optionally a plasticizer, optionally an ultraviolet protector, optionally an activity enhancer, optionally a glidant, and water, provided that the pH of the aqueous mixture is below the solubilization pH of the pH-dependent polymer; and

b) drying the aqueous mixture of step (a) to produce a coated pesticidal matrix.

Advantageously, it has been found that coated pesticidal matrices, prepared from a pH-dependent polymer without converting a substantial number of free carboxylic acid groups in the polymer to their salt form, retain a high percentage of their original activity after exposure to ultraviolet radiation and have greater residual activity compared to coated pesticidal agents prepared by the coating process described in EP 697170-A1. The instant process accomplishes this by providing an aqueous mixture wherein the pH is below the solubilization pH of the pH-dependent polymer.

In a preferred embodiment of the present invention, coated pesticidal matrices prepared by the process of this invention comprise about 1 to 50% by weight of a pesticidal agent, about 5 to 50% by weight of a pH-dependent polymer, 0 to about 25% by weight of a plasticizer, 0 to about 30% by weight of an ultraviolet protector, 0 to about 75% by weight of an activity enhancer, and 0 to about 15% by weight of a glidant.

More preferred coated pesticidal matrices prepared by the process of this invention are those comprising about 5 to 35% by weight of a pesticidal agent, about 10 to 45% by weight of a pH-dependent polymer, 0 to about 25% by weight of a plasticizer, 0 to about 20% by weight of an ultraviolet protector, 0 to about 45% by weight of an activity enhancer, and 0 to about 10% by weight of a glidant.

The aqueous mixture of this invention may be dried using any conventional drying technique which allows the pH-dependent polymer to form a coating film on the outside, and a binding film inside, of the matrix particles. Preferably, the aqueous mixture is spray dried or air dried. The coated pesticidal matrices of

the present invention preferably have a particle size less than about 20 μm, and, more preferably, have a particle size of about 2 μm to 10 μm.

Pesticidal agents suitable for use in the present invention include chemical and biological insecticides, acaricides, nematicides, fungicides, herbicides, and the like, and mixtures thereof. In particular, pesticidal agents which are subject to inactivation of their desired activity by ultraviolet radiation are preferred pesticidal agents for use in this invention.

Chemical insecticides include, but are not limited to, arylpyrroles such as chlorfenapyr; amidinohydrazones such as hydramethylnon; hydrazinecarboxyamides such as those described in U.S. 5,543,573; 1,4-diaryl-2-fluoro-2-butenes such as those described in EP 811593-A1, including 1-[1-(*p*-chlorophenyl)-2-fluoro-4-(4-fluoro-3-phenoxyphenyl)-2-butenyl]cyclopropane, (R,S)-(Z)-; 1-substituted-2-(nitromethylene)imidazolidines such as imidacloprid and 1-(6-chloro-3-pyridyl)-2-(nitromethylene)imidazolidine; phenylpyrazoles such as fipronil; and the like, and mixtures thereof. The chemical insecticides of this invention, when in solid form, preferably have a particle size prior to coating of less than about 10 μm and, more preferably, have a particle size of about 0.1 μm to 5 μm.

Biological insecticides include all naturally occuring and genetically modified varieties of insect biological control agents such as viral pathogens, bacterial pathogens, and fungal pathogens. Viral pathogens suitable for use include DNA viruses, RNA viruses and unclassified insect viruses such as gonad-specific virus (GSV).

The DNA viruses include double stranded enveloped DNA viruses such as (Subfamily, then species)

Entomopoxvirinae (*Melolontha melolontha* entomopoxvirus), and *Eubaculovirinae* (*Autographa californica* MNPV; *Heliocoverpa zea* NPV; *Trichoplusia ni* GV), as well as double stranded nonenveloped DNA viruses such as *Iridoviridae* (*Chilo* iridescent virus) and single stranded nonenveloped DNA viruses such as *Parvoviridae* (Galleria densovirus).

The RNA viruses include double stranded enveloped RNA viruses such as *Togaviridae* (Sindbis virus), *Bunyaviridae* (Beet leafcurl virus) and *Flaviviridae* (Wesselbron virus), as well as double stranded nonenveloped RNA viruses such as *Reoviridae* (Corriparta virus) and *Birnaviridae* (Drosophila X virus), as well as single stranded nonenveloped RNA viruses such as *Picornaviridae* (Cricket paralysis virus), *Tetraviridae* (*Heliothis armigera* stunt virus) and *Nodaviridae* (Black beetle virus).

The Subfamily of double stranded DNA viruses *Eubaculovirinae* includes two genera, nuclear polyhedrosis viruses (NPVs) and granulosis viruses (GVs), which are particularly useful for biological control because they produce occlusion bodies in their life cycle. Examples of NPVs include *Lymantria dispar* NPV (gypsy moth NPV); *Autographa californica* NPVs such as V8vEGTDEL, V8vEGTDEL-AaIT, AcMNPV E2, AcMNPV L1, AcMNPV V8, and AcMNPV Px1; *Anagrapha falcifera* NPV (celery looper NPV); *Spodoptera littoralis* NPV; *Spodoptera frugiperda* NPV; *Heliothis armigera* NPV; *Mamestra brassicae* NPV; *Choristoneura fumiferana* NPV; *Trichoplusia ni* NPV; *Heliocoverpa zea* NPV; and *Rachiplusia ou* NPV; and the like. Examples of GVs include *Cydia pomonella* GV (coddling moth GV), *Pieris*

brassicae GV, *Trichoplusia ni* GV, *Artogeia rapae* GV, *Plodia interpunctella* GV (Indian meal moth), and the like. Examples of entomopox viruses (EPVs) include *Melolontha melolontha* EPV, *Amsacta moorei* EPV, *Locusta migratoria* EPV, *Melanoplus sanguinipes* EPV, *Schistocerca gregaria* EPV, *Aedes aegypti* EPV, *Chironomus luridus* EPV, and the like.

Bacterial pathogens suitable for use include, but are not limited to, *Bacillus thuringiensis*, *Bacillus lentimorbus*, *Bacillus cereus*, *Bacillus popilliae*, *Photorhabdus luminescens*, *Xenorhabdus nematophilus*, and the like. Fungal pathogens suitable for use include, but are not limited to, *Beauveria bassiana*, *Entomophthora spp.*, *Metarrhizium anisopliae*, and the like.

AcMNPV E2 is described in EP 621337, and co-pending U.S. Serial No. 08/009,264, filed January 25, 1993, which is incorporated herein by reference. AcMNPV V8 and V8vEGTDEL are described in U.S. Patent 5,662,897 which is incorporated herein by reference. V8vEGTDEL-AaIT is described in EP 697170-A1 and co-pending U.S. Serial No. 08/322,679, filed July 27, 1994. AcMNPV Px1 is described in co-pending provisional U.S. Serial No.60/084,705, filed May 8, 1998, which is incorporated herein by reference.

Herbicides suitable for use in the present invention include chemical and biological herbicides. Chemical herbicides include, but are not limited to, dinitro-anilines such as pendimethalin and trifluralin; imidazolinones such as imazethapyr, imazaquin, imazamethabenz-methyl, imazapyr, imazamox and imazapic; haloacetanilides such as alachlor, metolachlor, and propachlor; and the like; and mixtures thereof. Biological herbicides include, but are not limited to,

fungal pathogens such as a *Dactylaria higginsii*, and the like, and mixtures thereof.

pH-Dependent polymers suitable for use in the present invention include polymers which are essentially insoluble below about pH 5.5, such as ethyl acrylate/methacrylic acid copolymers, methyl methacrylate/methacrylic acid copolymers, methacrylic acid/methyl acrylate/methyl methacrylate copolymers and the like, and mixtures thereof. Preferred pH-dependent polymers include ethyl acrylate/methacrylic acid copolymers wherein the ratio of free carboxyl groups to esters is about 1:1 (Eudragit® L 30 D, solubilization pH > 5.5, available from Röhm Pharma GmbH, Weiterstadt, Germany; and Kollicoat® MAE 30 D, solubilization pH > 5.5, available from BASF, Ludwigshafen, Germany), methyl methacrylate/methacrylic acid copolymers wherein the ratio of free carboxyl groups to esters is from about 1:1 to about 1:2 (Eudragit® S100, 1:2 ratio, solubilization pH > 7.0, available from Röhm Pharma; and Eudragit® L100, 1:1 ratio, solubilization pH > 6.0, available from Röhm Pharma), methacrylic acid/methyl acrylate/methyl methacrylate copolymers wherein the ratio of methacrylic acid, methyl acrylate and methyl methacrylate monomers is about 1:5:2 to 3:7:3 (Preparation 4110D, 1:6.5:2.5 ratio, solubilization pH > 7.2, available from Röhm Pharma), and mixtures thereof.

The pH-dependent polymer should be essentially insoluble below about pH 5.5 to prevent premature release of the pesticide when the coated pesticidal matrix is applied to the locus of a pest. In addition, when the pesticidal agent is an insecticide, the pH-dependent polymer should be soluble in the environment of the insect's gut so that the pesticidal agent may be readily released from the coated pesticidal matrix. Preferably, the pH-dependent polymer should be soluble above about pH

7 to ensure that the pesticide is readily released in the insect's gut.

In a preferred embodiment of the process of this invention, the methyl methacrylate/methacrylic acid copolymer is partially solubilized with base to reduce agglomeration of the copolymer particles prior to drying. However, it should be understood that the amount of base added is well below the amount required to fully solubilize the copolymer. Typically, less than about 10% of the free carboxylic acid groups of the copolymer are converted to salts. Bases suitable for use to partially solubilize the methyl methacrylate/methacrylic acid copolymers of this invention include ammonium hydroxide, alkali metal hydroxides, alkaline earth metal hydroxides and the like, with ammonium hydroxide being preferred.

Plasticizers are used in the process of this invention to reduce the minimum film forming temperature of the pH-dependent polymer. Plasticizers suitable for use in the present invention include any of the conventional agents known in the art such as poly(ethylene glycols), poly(propylene glycols), diethyl phthalate, dibutyl phthalate, citric acid esters such as triethyl citrate and the like, castor oil, triacetin and the like or mixtures thereof. Preferred plasticizers include poly(ethylene glycols) having an average molecular weight of about 1,000 to 10,000 and triethyl citrate.

Ultraviolet protectors are used in the present invention to reduce the photoinactivation of the pesticidal agent. Ultraviolet protectors suitable for use include ultraviolet absorbers and ultraviolet reflectors or mixtures thereof. Ultraviolet absorbers include various forms of carbon, such as carbon black (charcoal); benzophenones, such as 2-hydroxy-4-methoxybenzophenone (CYASORB® UV9, available from Cytec

Industries, West Paterson, New Jersey), 2,2'-dihydroxy-4-methoxybenzophenone (CYASORB® UV24, available from Cytec Industries), 2-hydroxy-4-acryloyloxyethoxybenzophenone (CYASORB® UV2098, available from Cytec Industries), 2-hydroxy-4-n-octoxybenzophenone (CYASORB® UV531, available from Cytec Industries); dyes, such as congo red, malachite green, malachite green hydrochloride, methyl orange, methyl green, brilliant green, acridine yellow, FDC green, FDC yellow, FDC red, and the like. Ultraviolet reflectors include titanium dioxide and the like. Preferred ultraviolet protectors include carbon black, benzophenones, dyes and titanium dioxide; with titanium dioxide, carbon black, CYASORB® UV9 and CYASORB® UV24 being most preferred.

Activity enhancers are used in this invention to enhance pesticidal activity of the pesticidal agent. Activity enhancers suitable for use in this invention include fluorescent brighteners described in U.S. Patent 5,124,149 and stilbene compounds described in U.S. Patent 5,246,936, both incorporated herein by reference. In addition to enhancing pesticidal activity, the stilbene compounds also provide some protection from ultraviolet radiation. Preferred stilbene compounds are the analogues of 4,4'-diamino-2,2'-stilbene disulfonic acid, namely, a Calcofluor White (available from Sigma Chemical Co., St. Louis, Missouri) such as Calcofluor White M2R, Calcofluor White ABT, Calcofluor White LD, Calcofluor White RWP, etc.; a Blancophor (available from Mobay Chemicals, Pittsburgh, Pennsylvania) such as Blancophor BBH, Blancophor MBBH, Blancophor BHC, etc.; an INTRAWITE® (a heterocyclic stilbene derivative, available from Crompton and Knowles Corp., Charlotte, North Carolina) such as INTRAWITE® CF, etc.; a Leucophor (available from Sandoz Chemicals Corp., Charlotte, North Carolina) such as Leucophor BS, Leucophor BSB, Leucophor EKB, Leucophor

PAB, etc.; a Phorwite (available from Mobay Chemicals) such as Phorwite AR, Phorwite BBU, Phorwite BKL, Phorwite CL, Phorwite RKK, etc. and the like. Blancophor BBH, Calcofluor White M2R and Phorwite AR are the most preferred stilbene compounds.

Glidants are used in the process of this invention to keep the dried, coated pesticidal matrix particles from sticking together. In addition, the glidant may also provide some protection from ultraviolet radiation. Glidants suitable for use in this invention include talc, magnesium stearate, calcium stearate, calcium sulfate and the like or mixtures thereof, with talc being preferred.

Other compatible additives such as preservatives, stabilizers (trehalose), anti-foam agents, anti-mold agents, anti-fungal agents, anti-bacterial agents and the like may also be included in the matrices of the present invention. Clearly, anti-fungal agents and anti-bacterial agents generally would not be used when fungal pathogens and bacterial pathogens, respectively, are used.

The present invention also provides wettable powder pesticidal compositions which comprise about 0.5 to 40% by weight of a dispersing agent; about 1 to 10% by weight of a flow enhancing agent; about 10 to 70% by weight of a bulking agent; 0 to about 25% by weight of a wetting agent; 0 to about 35% by weight of a pH-modifying agent; and about 5 to 75% by weight of a coated pesticidal matrix prepared by the process of this invention.

Preferred wettable powder pesticidal compositions of the present invention are those comprising about 2 to 15% by weight of a dispersing agent; about 1 to 10% by weight of a flow enhancing agent; about 10 to 60% by weight of a bulking agent; 0 to about 15% by weight of a wetting agent; 0 to about 20% by weight of a pH-modifying agent;

and about 5 to 75% by weight of a coated pesticidal matrix prepared by the process of this invention.

When the pesticidal agent is a biological agent, the wettable powder compositions of this invention preferably comprise about 2 to 10% by weight of a dispersing agent; about 1 to 10% by weight of a flow enhancing agent; about 20 to 50% by weight of a bulking agent; about 2 to 20% by weight of a pH-modifying agent; and about 15 to 60% by weight of a coated biological agent matrix prepared by the process of this invention.

Dispersing agents useful in the wettable powder pesticidal compositions of this invention include any of the conventional agents known in the art. Preferred dispersing agents are anionic agents, such as salts of the condensation products of formaldehyde with the sulfonation products of polycyclic aromatic compounds, sodium lignosulfonate and the like or mixtures thereof with the sodium sulfonate of naphthalene formaldehyde condensates such as MORWET® D425 (available from Witco), LOMAR® PW (available from Henkel, Ambler, Pennsylvania) and DARVAN® 1 (available from R.T. Vanderbilt Co., Norwalk, Connecticut) being most preferred.

Flow enhancing agents useful in the wettable powder pesticidal compositions of this invention include conventional flow enhancing agents known in the art with silicates such as calcium silicates being preferred. MICRO-CEL® E (a synthetic calcium silicate hydrate available from Celite Corp., Lompoc, California) is the most preferred flow enhancing agent.

Bulking agents suitable for use in the compositions of the present invention include natural and synthetic clays and silicates, e.g., natural silicas such as diatomaceous earths; magnesium silicates such as talcs; magnesium aluminum silicates such as attapulgites and vermiculites; aluminum silicates such as kaolinites,

montmorillonites and micas; and hydrated aluminum silicates such as kaolin clay. Preferred bulking agents are hydrated aluminum silicates, aluminum silicates, magnesium silicates and magnesium aluminum silicates, with kaolin clay being the most preferred bulking agent.

Wetting agents suitable for use in the present invention include any of the conventional agents known in the art. Preferred wetting agents include anionic agents such as sodium N-methyl-N-oleoyltaurate, octylphenoxy polyethoxy ethanol, nonylphenoxy polyethoxy ethanol, sodium dioctyl sulfosuccinate, sodium dodecyl benzene sulfonate, sodium lauryl sulfate, sodium alkyl naphthalene sulfonate, sodium sulfonated alkyl carboxylate and the like or mixtures thereof. A mixture of sodium alkyl naphthalene sulfonate and sodium sulfonated alkyl carboxylate (MORWET® EFW available from Witco, Houston, Texas) is a highly preferred wetting agent.

pH-Modifying agents are used to maintain the pH of aqueous tank-mixes prepared from the compositions of this invention below about pH 5. pH-Modifying agents suitable for use include, but are not limited to, potassium hydrogen phthalate, and solid organic acids such as citric acid, glutamic acid, maleic acid, *d,l*-malic acid, glutaric acid, isophthalic acid, succinic acid, fumaric acid, adipic acid, and the like, and mixtures thereof. Citric acid is especially useful as the pH-modifying agent in the compositions of this invention. In the compositions of this invention, it is preferable to use a granular organic acid having a mean particle size greater than about 50 μm, preferably greater than about 100 μm. The use of a granular organic acid improves the storage stability of the wettable powder compositions of this

invention when compared to wettable powder compositions containing a micronized organic acid.

The wettable powder pesticidal compositions of the present invention are typically prepared by blending a mixture of a dispersing agent, a bulking agent, a flow enhancing agent, optionally a wetting agent and optionally a pH-modifying agent to form a premix. This premix is then blended with the coated pesticidal matrix to form the desired wettable powder pesticidal compositions of the present invention.

For the control of pests, the wettable powder pesticidal compositions of this invention are diluted with water to form an aqueous tank-mix and the tank-mix is applied to the locus of the pest.

Surprisingly, it has been discovered that the coated pesticidal matrices of this invention provide improved residual control of pests when compared to coated pesticidal agents prepared according to the aqueous coating process described in EP 697170-A1. Accordingly, the present invention provides a method for improving the residual control of a pest by applying to the locus of the pest a pesticidally effective amount of a coated pesticidal matrix prepared by the process of this invention.

Other ingredients such as attractants, stickers, anti-foaming agents and the like may also be added to the wettable powder compositions of this invention. However, those additional ingredients are generally added separately to the tank-mix. An adjuvant or mixture of adjuvants may also be added to the tank-mix.

In order to facilitate a further understanding of the invention, the following examples are presented primarily for the purpose of illustrating more specific details thereof. The invention should not be deemed limited thereby except as defined in the claims.

EXAMPLE 1

Preparation of coated pesticidal matrices using an ethyl acrylate/methacrylic acid copolymer

A mixture of V8vEGTDEL polyhedral inclusion bodies (PIBs) (12.43 g of technical material, 7.5 g of PIBs, about 1.27×10^{11} PIBs/gram, mean PIB size about 2.5 μm), water (65.02 g), Blancophor BBH (28.04 g, mean particle size about 1 μm), PEG 5000 (poly(ethylene glycol) average MW 5000, 14.0 g of a 10 wt/wt% solution), and Kollicoat® MAE 30 D (46.71 g) is stirred to obtain a slurry. The slurry is filtered through an 80 mesh screen and spray dried using a Büchi spray drier (model 190) to obtain the coated pesticidal matrix identified as composition 1 in Table II.

Using essentially the same procedure, but using the ingredients listed in Table I, the coated pesticidal matrices identified as compositions 2-17 in Table II are prepared.

TABLE I

Pesticidal Agent

a. V8vEGTDEL polyhedral inclusion bodies
b. V8vEGTDEL-AaIT polyhedral inclusion bodies
c. Hydramethylnon
d. *Bacillus thuringiensis*

Ethyl Acrylate/Methacrylic Acid Copolymer

e. Kollicoat® MAE 30 D
f. Eudragit® L 30 D

Plasticizer

g. PEG 5000
h. PEG 8000

Stilbene Compound

i. Blancophor BBH
j. Calcofluor M2R

UV-Protector

k. Titanium dioxide
l. Charcoal

Additional Compound

m. Antifoam A® (a polydimethylsiloxane and silica antifoam agent available from Dow Corning, Midland, Michigan)

TABLE II

Pesticidal Matrices

Composition	Ingredient / wt/wt%					
	Pesticidal Agent	Copolymer	Plasticizer	Stilbene Compound	UV - Protector	Additional Compound
1	a/11.25	e/28.62	g/2.86	i/57.27	-	-
2	a/12.23	f/23.30	g/2.33	i/62.14	-	-
3	a/12.25	f/18.37	-	i/69.39	-	-
4	a/16.19	f/20.96	h/1.19	i/61.65	-	-
5	a/20.38	f/26.38	h/1.50	-	k/51.73	-
6	a/33.31	f/43.11	h/2.45	j/21.13	-	-
7	a/16.00	f/20.71	h/2.36	i/60.93	-	-
8	a/13.30	f/17.22	h/1.96	i/25.32	k/42.20	-
9	a/20.55	f/25.74	h/1.49	-	k/52.22	-

[1] Compositions may contain a small amount of residual water.

TABLE II (cont.)

Composition	Ingredient / wt/wt%[1]					
	Pesticidal Agent	Copolymer	Plasticizer	Stilbene Compound	UV - Protector	Additional Compound
10	a/16.11	f/20.44	g/2.05	i/61.40	-	-
11	a/12.22	f/23.29	g/2.33	i/62.16	-	-
12	a/13.90	f/26.52	g/2.62	i/49.93	l/7.02	-
13	a/21.28	f/19.17	g/1.92	i/57.64	-	-
14	b/14.16	f/27.05	g/2.13	i/56.66	-	-
15	b/15.06	e/22.77	g/2.23	i/59.94	-	-
16	c/13.95	f/27.91	g/2.33	i/55.81	-	-
17	d/14.15	f/27.02	g/2.06	i/56.61	-	m/0.16

EXAMPLE 2

Preparation of coated pesticidal matrices using a methyl methacrylate/methacrylic acid copolymer

A slurry is prepared by sequentially mixing V8vEGTDEL polyhedral inclusion bodies (13.0 g of technical material, 6.0 g of PIBs, about 1.27 x 10^{11} PIBs/gram, mean PIB size about 2.5 μm), water, 56.6 g of a copolymer slurry (previously prepared by mixing Eudragit® S100 (30.0 g), water (166 g), 1 N ammonium hydroxide solution (15.24 g) and triethyl citrate (15.0 g)), Blancophor BBH (14.0 g), talc (3.21 g), charcoal (9.0 g), a solution of Calcofluor M2R (14.0 g) in water, and water. The resultant slurry is then filtered through an 80 mesh screen and spray dried using a Büchi spray drier (model 190) to obtain the coated pesticidal matrix identified as composition 18 in Table IV.

Using essentially the same procedure, but using the ingredients listed in Table III, the coated pesticidal matrices identified as compositions 19-26 in Table IV are prepared.

TABLE III

Pesticidal Agent

a. V8vEGTDEL polyhedral inclusion bodies
b. V8vEGTDEL-AaIT polyhedral inclusion bodies
c. Yeast

Methyl Methacrylate/Methacrylic Acid Copolymer

d. Eudragit® S100
e. Eudragit® L100

Plasticizer

f. Triethyl citrate

UV-Protector

g. Charcoal

Stilbene Compound

h. Blancophor BBH
i. Calcofluor M2R

Glidant

j. Talc

Additional Compound

k. Citric acid
l. Microat® afa Complex (an antioxidant available from Nurture Inc., Missoula, Montana)

TABLE IV

Pesticidal Matrices

Composition	Ingredient / wt/wt%[1]						
	Pesticidal Agent	Copolymer	Plasticizer	UV-Protector	Stilbene Compound	Glidant	Additional Compound
18	a/11.89	d/14.86	f/7.43	g/17.84	h/27.75 i/13.87	j/6.36	-
19	a/11.89	d/14.86	f/7.43	g/17.84	h/35.08 i/ 6.54	j/6.36	-
20	a/12.36	e/15.45	f/7.72	g/18.54	h/37.08	j/7.72	k/1.13
21	a/12.12	e/15.15	f/7.58	g/18.18	h/36.36	j/7.58	k/1.01 l/2.02
22	b/10.90	d/16.59	f/8.30	g/16.59	h/38.06 i/4.36	j/5.20	-
23	b/8.68	d/17.59	f/8.81	g/16.77	h/38.47 i/4.41	j/5.26	-
24	b/8.77	d/16.94	f/8.47	g/17.20	h/38.86 i/4.45	j/5.31	-
25	a/10.90	d/16.59	f/8.30	g/16.59	h/38.06 i/4.36	j/5.20	-
26	c/21.61	e/40.36	-	-	h/38.04	-	-

[1] Compositions may contain a small amount of residual water.

EXAMPLE 3

Preparation of coated pesticidal matrices using a methacrylic acid/methyl acrylate/methyl methacrylate copolymer

A mixture of chlorfenapyr (3.00 g, mean particle size about 2.5 μm), water (100.00 g), Blancophor BBH (12.00 g, mean particle size about 1 μm), triethyl citrate (0.23 g), a 20% solution of Preparation 4110D (22.50 g), talc (3.00 g), and MORWET®D425 (1.50g) is stirred to obtain a slurry. The slurry is filtered through an 80 mesh screen and spray dried using a Büchi spray drier (model 190) to obtain the coated pesticidal matrix identified as composition 27 in Table VI.

Using essentially the same procedure, but using the ingredients listed in Table V, the coated pesticidal matrices identified as compositions 28-32 in Table VI are prepared.

TABLE V

Pesticidal Agent

a. Chlorfenapyr

b. Hydramethylnon

c. 1-(6-Chloro-3-pyridyl)-2-(nitromethylene)imidazolidine

d. V8vEGTDEL-AaIT polyhedral inclusion bodies

Methacrylic Acid/Methyl Acrylate/Methyl Methacrylate Copolymer

Preparation 4110D

Plasticizer

Triethyl citrate

Stilbene Compound

e. Blancophor BBH

f. Calcofluor M2R

UV-Protector

Charcoal

Glidant

Talc

Additional Compound

MORWET®D425

TABLE VI

Pesticidal Matrices

Composition	Pesticidal Agent	Preparation 4110D	Triethyl Citrate	Stilbene Compound	Charcoal	Talc	MORWET®D425
				Ingredient / wt/wt%[1]			
27	a/12.38	18.57	0.95	e/49.53	-	12.38	6.19
28	a/9.01	18.02	0.90	e/36.04 f/9.01	13.51	9.01	4.50
29	b/12.38	18.57	0.95	e/49.53	-	12.38	6.19
30	b/9.01	18.02	0.90	e/36.04 f/9.01	13.51	9.01	4.50
31	c/13.97	21.12	1.05	e/42.76	-	13.97	7.13
32	d/9.36	18.71	1.87	e/37.42 f/4.57	18.71	9.36	-

[1]Compositions may contain a small amount of residual water.

EXAMPLE 4

Preparation of a coated pesticidal matrix using a methyl methacrylate/methacrylic acid copolymer, REAX® 85A and Indulin® C

A mixture of V8vEGTDEL polyhedral inclusion bodies (13.0 g of technical material, 6.0 g of PIBs, about 1.27 x 10^{11} PIBs/gram, mean PIB size about 2.5 μm) and ammonium hydroxide solution (15.0 g, pH 9.5) is stirred for 15 minutes, treated with REAX® 85A (0.18 g, a sodium lignosulfonate available from Westvaco, Charleston Heights, South Carolina), stirred for 15 minutes, treated with Indulin® C (12.0 g of a 2% solution, pH 11, a sodium lignate available from Westvaco), stirred for one hour, and adjusted slowly to pH 4.5 with dilute sulfuric acid over 2.5 hours. After stirring for 45 minutes, the polyhedral inclusion body mixture is mixed with the copolymer slurry described in Example 2 (56.6 g), Blancophor BBH (14.70 g), talc (3.21 g), charcoal (9.0 g), a solution of Calcofluor M2R (3.30 g) in water, and water to obtain a slurry. The slurry is filtered through an 80 mesh screen and spray dried using a Büchi spray drier (model 190) to obtain the coated pesticidal matrix identified as composition 33 in Table VII.

TABLE VII

Composition 33

Ingredient	wt/wt%
V8vEGTDEL polyhedral inclusion bodies	12.53
Eudragit® S100	15.66
Triethyl citrate	7.83
Charcoal	18.80
Blancophor BBH	30.70
Calcofluor M2R	6.89
Talc	6.70
Indulin® C	0.50
REAX® 85A	0.38

EXAMPLE 5

Preparation of wettable powder pesticidal compositions

The coated pesticidal matrix identified as composition 18 in Table IV (23.13 g) is added to a premix of MORWET® EFW (3.84 g), MORWET® D425 (7.68 g), kaolin clay (23.03 g), MICRO-CEL® E (2.30 g), and citric acid (11.52 g). The resultant mixture is blended to obtain the wettable powder composition identified as composition 34 in Table VIII.

Using essentially the same procedure, the wettable powder compositions identified as compositions 35-53 in Table VIII are prepared.

TABLE VIII
Wettable Powder Pesticidal Compositions

	Ingredient / wt/wt%					
Composition	Coated Pesticidal Matrix[1]	MORWET® EFW	MORWET® D425	Kaolin Clay	MICRO-CEL®E	Citric Acid
34	18/32.35	5.37	10.74	32.21	3.22	16.11[2]
35	1/30.77	5.50	10.99	32.97	3.30	16.48[2]
36	2/25.91	5.88	11.76	35.29	3.53	17.63[2]
37	9/16.05	6.67	13.32	39.98	4.00	19.99[2]
38	11/25.91	5.88	11.76	35.28	3.53	17.64[2]
39	12/16.98	7.56	15.11	49.81	4.98	5.56[2]
40	13/19.80	6.37	12.73	38.20	3.82	19.09[2]
41	15/21.52	7.13	14.30	47.10	4.70	5.25[2]
42	19/32.35	5.37	10.74	32.21	3.22	16.11[2]
43	20/31.12	5.47	10.94	32.79	3.28	16.40[2]
44	21/31.73	5.42	10.84	32.50	3.25	16.25[2]

[1] The coated pesticidal matrix is identified by the composition number from Tables II, IV or VI.
[2] Mean particle size about 1-3 µm.
[3] Mean particle size greater than about 100 µm.

TABLE VIII (Cont.)

	Ingredient / wt/wt%					
Composition	Coated Pesticidal Matrix[1]	MORWET® EFW	MORWET® D425	Kaolin Clay	MICRO-CEL®E	Citric Acid
45	22/30.70	5.50	11.00	32.99	3.30	16.50[2]
46	22/33.47	5.70	11.40	36.16	3.61	9.66[2]
47	22/32.79	5.34	10.67	32.01	3.20	16.00[2]
48	23/31.94	5.52	11.04	36.39	3.64	11.47[2]
49	25/33.86	6.02	12.04	39.68	3.97	4.43[2]
50	27/43.86	5.11	10.22	33.68	3.37	3.76[2]
51	28/60.42	3.60	7.20	23.75	2.37	2.65[2]
52	31/38.76	5.00	10.00	32.94	3.30	10.00[3]
53	32/42.80	-	3.00	37.20	6.00	11.00[3]

[1] The coated pesticidal matrix is identified by the composition number from Tables II, IV or VI.
[2] Mean particle size about 1-3 µm.
[3] Mean particle size greater than about 100 µm.

EXAMPLE 6

Evaluation of wettable powder pesticidal compositions of this invention and a wettable powder pesticidal composition disclosed in EP 697170-A1 against tobacco budworms

Wettable powder compositions 34, 36, 37 and 43, and a control composition, identified below, are tested for efficacy against neonate tobacco budworms, *H. virescens*, on cotton variety IAC-22 through bioassay of field-treated foliage. Each composition is mixed with water, 0.2 w/v% KINETIC® (nonionic surfactant mixture available from Helena Chemical Co., Memphis, Tennessee), and 3.5 w/v% MIRASPERSE® (2-hydroxypropyl ether starch available from A.E. Staley Manufacturing Co., Decatur, Illinois). In addition, 0.1 w/v% citric acid is added to the aqueous control composition. Treatments are applied with a CO_2 backpack sprayer calibrated to deliver 200 L/ha using 2 ft boom with hollow-cone nozzles (3/row; 1 centered and 2 drop).

For bioassay, leaves are collected 1-2 hours following application for initial activity and 1, 2, 3, and 4 days after treatment for residual activity. The treated leaves are placed in petri dishes with moist filter papers (1 leaf/dish; 4 larvae/dish; 16 dishes/-treatment with a total of 64 larvae/treatment/sampling period). After allowing the larvae to feed on the treated leaves for four days, they are transferred to diet trays containing pieces of untreated cotton leaves; one larva/cell. After 4 days, the surviving larvae are counted. The results are summarized in Table IX.

As can be seen from the data in Table IX, compositions containing coated pesticidal matrices prepared by the process of the present invention, in general, have greater residual activity against

H. virescens than the control composition prepared by the aqueous process described in EP 697170-A1. In particular, composition 34 has significantly greater residual activity than the control composition. This is an especially surprising discovery because the copolymer used in composition 34 and the control composition is the same Eudragit® S100.

Control Composition

Ingredient	wt/wt%
[1]Coated pesticidal agent	25.14
MORWET® EFW	5.94
MORWET® D425	11.89
Kaolin Clay	35.64
MICRO-CEL® E	3.56
Citric Acid	17.83

[1]Prepared according to the aqueous process described in EP 697170-A1. The coated pesticidal agent contains 15.31 wt/wt% V8vEGTDEL polyhedral inclusion bodies, 15.31 wt/wt% Eudragit® S100, 0.43 wt/wt% PEG 8000, 23.04 wt/wt% charcoal and 45.92 wt/wt% Blancophor BBH.

TABLE IX

Percent Mortality of *H. virescens* on Cotton variety IAC-22

	Days After Treatment				
Treatment	0	1	2	3	4
Composition 34	100	92	95	89	75
Composition 36	98	97	77	75	66
Composition 37	97	94	92	77	67
Composition 43	98	95	95	86	80
Control composition	95	86	83	73	69
Untreated	5	2	5	8	6

EXAMPLE 7

Evaluation of wettable powder pesticidal compositions against tobacco budworms on cotton and lettuce

Compositions 39 and 49 from Table VIII are tested for efficacy against neonate tobacco budworms, *Heliothis virescens*, on lettuce variety Green-Towers and cotton variety Delta-Pine 51 through bioassay of field-treated foliage. The plots are strips of cotton and lettuce (ca. 40 ft long) with 3 ft row spacing. Each composition is mixed with water and applied at 8 x 10^{11} polyhedral inclusion bodies/acre. DIPEL® 2X (*Bacillus thuringiensis* var. Kurstaki, available from Abbott Laboratories, North Chicago, Illinois) is applied at 1.0 lb product/acre as a standard. Treatments are applied with a CO_2 backpack sprayer calibrated to deliver 20 gallons per acre using a 2 ft boom with hollow-cone nozzles (3/row; 1 centered and 2 drop).

For bioassay, leaves are collected 1-2 hours following application for initial activity and 2, 3, 4 and 5 days after treatment for residual activity. The treated leaves are placed in petri dishes with moist filter papers (1 leaf/dish; 4 larvae/dish; 16 dishes/-treatment with a total of 64 larvae/treatment/sampling period). After allowing the larvae to feed on the treated leaves for two days, they are transferred to diet trays; one larva/cell. The surviving larvae are also counted at 2, 4, 6 and 8 days after transfer to diet. The results are summarized in Tables X and XI.

As can be seen from the data in Tables X and XI, the pesticidal compositions of this invention (compositions 39 and 49) have greater residual activity against tobacco budworms after 4, 6 and 8 days on diet than DIPEL® 2X.

TABLE X

Percent Mortality of Tobacco Budworms on Cotton

Treatment	Days After Transfer to Diet	Days After Treatment				
		0	2	3	4	5
Composition 39	0	4	3	2	1	2
	2	24	9	6	3	4
	4	49	23	14	6	13
	6	50	27	16	7	15
	8	51	27	16	8	15
Composition 49	0	2	3	2	0	5
	2	52	17	9	7	6
	4	90	57	29	11	14
	6	91	59	31	15	16
	8	91	59	33	16	16
DIPEL® 2X	0	29	9	6	2	4
	2	37	13	7	4	6
	4	56	14	10	4	6
	6	56	14	12	5	6
	8	56	14	12	5	6
Untreated	0	1	2	1	1	2
	2	1	4	2	3	4
	4	2	6	3	3	5
	6	2	6	4	3	5
	8	2	6	4	3	5

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TABLE XI

Percent Mortality of Tobacco Budworms on Lettuce

Treatment	Days After Transfer to Diet	Days After Treatment				
		0	2	3	4	5
Composition 39	0	27	5	2	5	1
	2	72	36	16	20	11
	4	94	84	62	55	50
	6	95	85	67	59	52
	8	95	86	67	60	52
Composition 49	0	16	3	7	1	6
	2	73	24	23	18	16
	4	98	88	78	62	55
	6	99	88	85	62	58
	8	99	88	85	62	59
DIPEL® 2X	0	100	53	42	45	29
	2	100	70	50	57	35
	4	100	70	56	58	37
	6	100	70	56	58	38
	8	100	70	56	58	38
Untreated	0	2	1	2	2	4
	2	2	2	5	2	6
	4	4	5	6	3	9
	6	4	5	6	4	10
	8	4	5	6	6	10

EXAMPLE 8

Evaluation of non-irradiated and irradiated wettable powder compositions against *Heliothis virescens*

Plastic bioassay trays containing 32 open-faced wells (4 x 4 x 2.5 cm, L x W x H) per tray are utilized as test arenas in this evaluation. Five mL of Stoneville diet (soybean/wheat germ) is poured into each well and allowed to harden. Aqueous suspensions of the wettable powder pesticidal compositions are evenly spread over the surface of the hardened diet to provide 2 x 10^3 V8vEGTDEL polyhedral inclusion bodies per well. Half of the trays are placed under ultraviolet lamps (two FS40UVB bulbs set 30 cm above the trays, Atlantic Ultraviolet Corp., Bay Shore, NY) for four hours. All trays are then infested with one three-day-old *H. virescens* larva per well. The wells are covered with a vented, clear plastic sheet and held under constant fluorescent light at a temperature of about 27 °C. After ten days, the wells are examined and larval mortality measurements are made. The results are summarized in Table XII.

Advantageously, the wettable powder pesticidal compositions of this invention (composition numbers 35, 36, 38 and 40) retain at least 73 percent of their original activity after being exposed to ultraviolet light for 4 hours.

TABLE XII

Evaluation Of Non-Irradiated and Irradiated Wettable Powder Pesticidal Compositions Against *H. Virescens*

Wettable Powder Composition[1]	Irradiation Exposure (hours)	Percent Larval Mortality
35	0	98
	4	74
36	0	98
	4	80
38	0	97
	4	75
40	0	97
	4	71

[1] Composition number from Table VIII.

EXAMPLE 9

Field evaluation of wettable powder pesticidal compositions against tobacco budworms on tobacco in North Carolina

A field evaluation is conducted on tobacco grown near Clayton, North Carolina. A wettable powder composition of this invention (composition 46) at 2×10^{11}, 5×10^{11}, and 8×10^{11} bodies/acre, *Bacillus thuringiensis* (DIPEL® 2X, Abbott Laboratories) at 1.0 lb wettable powder (WP)/acre, and acephate (ORTHENE® 75SP, available from Valent USA, Walnut Creek, California) at 0.75 lb active ingredient (ai)/acre are compared for efficacy against *H. virescens*. Biological materials are suspended in water containing an insect gustatory stimulant (PHEAST® available from AGRISENSE, Fresno, California); aqueous dilutions of acephate contained no PHEAST®. Treatments and untreated check are replicated four times (small plots) in a randomized complete block design. By using fine-hair brushes, 1- to 2-day old laboratory-reared *H. virescens* are placed on the underside of leaves in each plot. Natural infestation of *H. virescens* also occurred at the test site. Treatments are applied to tobacco about 2 hours before each artificial larval infestation on days 1 and 8. Treatments are applied with a tractor-mounted, CO_2-pressurized boom sprayer which is calibrated to deliver 25 gallons/acre through a single D2-33 nozzle centered over each tobacco row. Boom pressure during application is 60 lb/in^2.

At 2 and 5 days after first application and 5 and 9 days after second application, live *H. virescens* are counted on 20 plants in each plot. Additionally, visual estimate of leaf damage caused by larval feeding is made

14 days after the second application using the rating scale shown below. The results are summarized in Table XIII.

Rating Scale

Rating	Meaning
4	Severe Damage
3	Heavy Damage
2	Moderate Damage
1	Slight Damage
0	No Damage

As can be seen from the data in Table XIII, the wettable powder composition of this invention (composition 46) provides good control of *H. virescens* on tobacco. In fact, on day 17 of the test, the invention composition provides greater control of *H. virescens* than DIPEL® 2X and acephate.

TABLE XIII

Field Evaluation of Wettable Powder Compositions - North Carolina

Treatment	Rate	Mean Number of Live Larvae per 20 Plants — Day of Test 3	8	13	17	Mean Plant Injury on Day 22 of Test
Composition 46	2 x 10^{11} bodies/acre	7.8	6.2	1.8	0.5	0.4
	5 x 10^{11} bodies/acre	6.8	6.2	1.2	0.2	0.3
	8 x 10^{11} bodies/acre	7.0	6.0	1.0	0.5	0.3
DIPEL® 2X	1.0 lb of WP/acre	5.5	5.0	1.2	2.5	0.1
Acephate	0.75 lb of ai/acre	6.0	5.8	1.2	0.8	0.2
Untreated		11.2	8.2	8.8	9.0	1.8

EXAMPLE 10

Field evaluation of wettable powder pesticidal compositions against tobacco budworms on tobacco in Georgia

A field evaluation is conducted on flue-cured tobacco (*var.* K-236) grown near Tifton, Georgia. A wettable powder composition of this invention (composition 47) at 2×10^{11}, 5×10^{11}, and 8×10^{11} bodies/acre, *Bacillus thuringiensis* (DIPEL® 4L, available from Abbott Laboratories) at 1.0 pint/acre, and methomyl (LANNATE® 2.4L, available from DuPont, Wilmington, Delaware) at 0.6 lb active ingredient (ai)/acre are compared for efficacy against *H. virescens*. Biological materials are suspended in water containing an insect gustatory stimulant (COAX® available from CCT Corp., Carlsbad, California) at 2.0 pints/acre; aqueous dilutions of methomyl contained no COAX®. Treatments and untreated check are replicated four times in a randomized complete block design. A treatment replicate consists of a five-row by 20 ft plot of tobacco. Treatments are applied to tobacco on days 1, 5, 9, 17 and 22 of the test. Treatments are applied with a backpack, CO_2-pressurized boom sprayer which is calibrated to deliver 20.7 gallons/acre through three TX12 (Spraying Systems, Wheaton, IL) hollow-cone nozzles per row (one nozzle above center of the row and one nozzle directed at each of the two sides of the row). Boom pressure during application is 40 lb/in^2.

On days 5, 8, 12, 22, 26 and 29 of the test, live *H. virescens* are counted on 20 plants in each plot. The results are summarized in Table XIV.

As can be seen from the data in Table XIV, the wettable powder composition of this invention (composition 47) provides good control of *H. virescens*.

EXAMPLE 11

Evaluation of UV stability of wettable powder compositions comprising chlorfenapyr

Wettable powder compositions 50 and 51 from Table VIII, and a control composition identified below are evaluated for UV stability. An aqueous suspension of each test composition is applied to plastic petri dishes (100 mm x 15 mm) using a belt sprayer with nozzles calibrated to provide 400 l/ha. The test materials are applied at rates to provide the equivalent of 0.5, 1.0 and 5.0 g of chlorfenapyr per hectare. The dishes are dried and exposed to UV light using either a UV-B lamp (280-315 nm) or natural light for various periods of time. Three second-instar tobacco budworm larvae (*Heliothis virescens*) are then placed in each dish and the dishes are covered. After holding the dishes at 26.7°C for 48 hours, the surviving larvae are counted. The results are summarized in Tables XV and XVI.

As can be seen from the data in Tables XV and XVI, chlorfenapyr treatments made with the wettable powder compositions of this invention are significantly more stable to UV exposure than the control composition which does not incorporate chlorfenapyr into a pesticidal matrix.

Control Composition

Ingredient	wt/wt%
Chlorfenapyr (tech.)	5.43
MORWET®EFW	8.60
MORWET®D425	17.21
Kaolin Clay	56.75
MIRO-CEL®E	5.67
Citric Acid[1]	6.34

[1]Mean particle size about 1-3 μm

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TABLE XV

Evaluation of UV (natural light) Irradiated Chlorfenapyr Wettable Powder Compositions

Wettable Powder Composition	Irradiation Exposure(days)	Percent Larval Mortality	
		1.0 g/ha	0.5 g/ha
50	0	100	86
	2	74	58
	3	28	34
51	0	100	91
	2	100	75
	3	63	48
Control Composition	0	100	97
	2	54	34
	3	15	22

TABLE XVI

Evaluation of UV-B Lamp Irradiated Chlorfenapyr Wettable Powder Compositions

Wettable Powder Composition	Irradiation Exposure(hours)	Percent Larval Mortality	
		5.0 g/ha	1.0 g/ha
50	0	100	100
	8	100	76
	22	100	76
	37	100	0
51	0	100	100
	8	100	47
	22	100	21
	37	56	0
Control Composition	0	100	100
	8	100	31
	22	100	24
	37	14	6

33343-01

WE CLAIM:

1. A process for the preparation of a coated pesticidal matrix which process comprises:

a) preparing an aqueous mixture comprising a pesticidal agent, a pH-dependent polymer, optionally a plasticizer, optionally an ultraviolet protector, optionally an activity enhancer, optionally a glidant, and water, provided that the pH of the aqueous mixture is below the solubilization pH of the pH-dependent polymer; and

b) drying the aqueous mixture of step (a) to produce the coated pesticidal matrix.

2. The process according to claim 1 wherein the plasticizer is present in the aqueous mixture.

3. The process according to claim 1 wherein the activity enhancer is present in the aqueous mixture.

4. The process according to claim 1 wherein the ultraviolet protector is present in the aqueous mixture.

5. The process according to claim 1 wherein the pesticidal agent is selected from the group consisting of an insecticide, an acaricide, a nematicide, a fungicide and a herbicide and mixtures thereof.

6. The process according to claim 5 wherein the insecticide agent is a chemical or a biological insecticide.

7. The process according to claim 6 wherein the chemical insecticide is selected from the group consisting of chlorfenapyr, hydramethylnon, imidacloprid, 1-(6-chloro-3-pyridyl)-2-(nitromethylene)imidazolidine, fipronil, and 1-[1-(*p*-chlorophenyl)-2-fluoro-4-(4-fluoro-3-phenoxyphenyl)-2-butenyl]cyclopropane, (R,S)-(Z)-, and mixtures thereof.

8. The process according to claim 6 wherein the biological insecticide is selected from the group consisting of V8vEGTDEL, V8vEGTDEL-AaIT, *Heliothis zea* NPV, *Lymantria dispar* NPV, AcMNPV E2, AcMNPV L1, ACMNPV V8, AcMNPV Px1, and *Bacillus thuringiensis*, and mixtures thereof.

9. The process according to claim 1 wherein the pH-dependent polymer is selected from the group consisting of an ethyl acrylate/methacrylic acid copolymer, a methyl methacrylate/methacrylic acid copolymer, a methacrylic acid/methyl acrylate/methyl methacrylate copolymer, and mixtures thereof; the plasticizer is selected from the group consisting of a poly(ethylene glycol), a poly(propylene glycol), a citric acid ester, diethyl phthalate, dibutyl phthalate, castor oil, triacetin, and mixtures thereof; the ultraviolet protector is selected from the group consisting of carbon black, a benzophenone, a dye, titanium dioxide, and mixtures thereof; the activity enhancer is a stilbene compound; and the glidant is selected from the group consisting of talc, magnesium stearate, calcium stearate, calcium sulfate, and mixtures thereof.

10. The process according to claim 9 wherein the pH-dependent polymer is selected from the group

consisting of an ethyl acrylate/methacrylic acid copolymer wherein the ratio of free carboxyl groups to esters is about 1:1, a methyl methacrylate/methacrylic acid copolymer wherein the ratio of free carboxyl groups to esters is from about 1:1 to about 1:2, a methacrylic acid/methyl acrylate/methyl methacrylate copolymer wherein the ratio of the monomers is about 1:5:2 to 3:7:3, and mixtures thereof; the plasticizer is selected from the group consisting of triethyl citrate and a poly(ethylene glycol) having an average molecular weight of about 1,000 to 10,000; and the stilbene compound is selected from the group consisting of Blancophor BBH, Calcofluor White M2R, Phorwite AR, and mixtures thereof.

11. The process according to claim 1 wherein the pH-dependent polymer is a methyl methacrylate/methacrylic acid copolymer and is partially solubilized with base.

12. The process according to claim 11 wherein the base is selected from the group consisting of ammonium hydroxide, an alkali metal hydroxide, and an alkaline earth metal hydroxide.

13. The process according to claim 1 wherein in said drying step the aqueous mixture is spray dried.

14. The process according to claim 1 wherein the coated pesticidal matrix has a particle size less than about 20 μm.

15. The process according to claim 14 wherein the coated pesticidal matrix has a particle size of about 2 μm to 10 μm.

16. The process according to claim 1 wherein the coated pesticidal matrix comprises about 1 to 50% by weight of the pesticidal agent, about 5 to 50% by weight of the pH-dependent polymer, 0 to about 25% by weight of the plasticizer, 0 to about 30% by weight of the ultraviolet protector, 0 to about 75% by weight of the activity enhancer, and 0 to about 15% by weight of the glidant.

17. A coated pesticidal matrix which comprises about 1 to 50% by weight of a pesticidal agent, about 5 to 50% by weight of a pH-dependent polymer wherein a substantial number of the free carboxylic acid groups in said polymer have not been converted to their salt form, 0 to about 25% by weight of a plasticizer, 0 to about 30% by weight of an ultraviolet protector, 0 to about 75% by weight of an activity enhancer, and 0 to about 15% by weight of a glidant.

18. The coated pesticidal matrix according to claim 17 which comprises about 5 to 35% by weight of the pesticidal agent, about 10 to 45% by weight of the pH-dependent polymer, 0 to about 25% by weight of the plasticizer, 0 to about 20% by weight of the ultraviolet protector, 0 to about 45% by weight of the activity enhancer, and 0 to about 10% by weight of the glidant.

19. The coated pesticidal matrix according to claim 17 wherein the pH-dependent polymer is selected from the group consisting of an ethyl acrylate/methacrylic acid copolymer, a methyl methacrylate/methacrylic acid copolymer, a methacrylic acid/methyl acrylate/methyl methacrylate copolymer, and mixtures thereof; the plasticizer is selected from the group consisting of a poly(ethylene glycol), a poly(propylene glycol), a citric

acid ester, diethyl phthalate, dibutyl phthalate, castor oil, triacetin, and mixtures thereof; the ultraviolet protector is selected from the group consisting of carbon black, a benzophenone, a dye, titanium dioxide, and mixtures thereof; the activity enhancer is a stilbene compound; and the glidant is selected from the group consisting of talc, magnesium stearate, calcium stearate, calcium sulfate, and mixtures thereof.

20. The coated pesticidal matrix according to claim 19 wherein the pH-dependent polymer is selected from the group consisting of an ethyl acrylate/methacrylic acid copolymer wherein the ratio of free carboxyl groups to esters is about 1:1, a methyl methacrylate/methacrylic acid copolymer wherein the ratio of free carboxyl groups to esters is from about 1:1 to about 1:2, a methacrylic acid/methyl acrylate/methyl methacrylate copolymer wherein the ratio of monomers is about 1:5:2 to 3:7:3, and mixtures thereof; the plasticizer is selected from the group consisting of triethyl citrate and a poly(ethylene glycol) having an average molecular weight of about 1,000 to 10,000; and the stilbene compound is selected from the group consisting of Blancophor BBH, Calcofluor White M2R, Phorwite AR, and mixtures thereof.

21. The coated pesticidal matrix according to claim 17 wherein the pesticidal agent is a chemical insecticide or a biological insecticide.

22. The coated pesticidal matrix according to claim 21 wherein the chemical insecticide is selected from the group consisting of chlorfenapyr, hydramethylnon, imidacloprid, 1-(6-chloro-3-pyridyl)-2-(nitromethylene)-imidazolidine, fipronil, and 1-[1-(*p*-chlorophenyl)-2-

fluoro-4-(4-fluoro-3-phenoxyphenyl)-2-butenyl]cyclopropane, (R,S)-(Z)-, and mixtures thereof.

23. The coated pesticidal matrix according to claim 21 wherein the biological insecticide is selected from the group consisting of V8vEGTDEL, V8vEGTDEL-AaIT, *Heliothis zea* NPV, *Lymantria dispar* NPV, AcMNPV E2, AcMNPV L1, ACMNPV V8, AcMNPV Px1, and *Bacillus thuringiensis*, and mixtures thereof.

24. The coated pesticidal matrix according to claim 17 having a particle size of less than about 20 μm.

25. The coated pesticidal matrix according to claim 24 having a particle size of about 2 μm to 10 μm.

26. A wettable powder pesticidal composition which comprises about 0.5 to 40% by weight of a dispersing agent; about 1 to 10% by weight of a flow enhancing agent; about 10 to 70% by weight of a bulking agent; 0 to about 25% by weight of a wetting agent; 0 to about 35% by weight of a pH-modifying agent; and about 5 to 75% by weight of a coated pesticidal matrix according to claim 17.

27. The composition according to claim 26 which comprises about 2 to 15% by weight of the dispersing agent; about 1 to 10% by weight of the flow enhancing agent; about 10 to 60% by weight of the bulking agent; 0 to about 15% by weight of the wetting agent; 0 to about 20% by weight of the pH-modifying agent; and about 5 to 75% by weight of the coated pesticidal matrix.

28. The composition according to claim 26 wherein the pesticidal agent in the coated pesticidal matrix is a biological agent.

29. The composition according to claim 28 which comprises about 2 to 10% by weight of the dispersing agent; about 1 to 10% by weight of the flow enhancing agent; about 20 to 50% by weight of the bulking agent; about 2 to 20% by weight of the pH-modifying agent; and about 15 to 60% by weight of the coated pesticidal matrix.

30. The composition according to claim 26 wherein the pH-modifying agent is an organic acid.

31. The composition according to claim 30 wherein the organic acid is citric acid.

32. The composition according to claim 30 wherein the organic acid has a mean particle size greater than about 50 μm.

33. The composition according to claim 32 wherein the organic acid has a mean particle size greater than about 100 μm.

34. A coated pesticidal matrix produced by the process of claim 1.

35. A method for improving the residual control of a pest which comprises applying to the locus of the pest a pesticidally effective amount of a coated pesticidal matrix according to claim 34.

33343-01

IMPROVED COATED PESTICIDAL MATRICES, A PROCESS FOR THEIR PREPARATION AND COMPOSITIONS CONTAINING THEM

ABSTRACT OF THE INVENTION

The present invention provides improved coated pesticidal matrices and a process for their preparation. The present invention also provides a wettable powder pesticidal composition containing the improved coated pesticidal matrices.

Docket No. 33343-01
Patent

COMBINED DECLARATION AND POWER OF ATTORNEY
(Original, Design, Supplemental, Divisional, Continuation,CIP)

As the below named inventor, I hereby declare that:

INVENTORSHIP IDENTIFICATION

My residence, post office address and citizenship are as stated below next to my name. I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled:

TITLE OF INVENTION

IMPROVED COATED PESTICIDAL MATRICES, A PROCESS FOR THEIR PREPARATION AND COMPOSITIONS CONTAINING THEM

SPECIFICATION IDENTIFICATION

the specification of which: (complete (a), (b), or (c))

(a) ☒ is attached hereto.

(b) ☐ was filed on as
- ☐ Serial Number
- ☐ Express Mail No. , as Serial Number not yet known

(c) ☐ was described and claimed in PCT International Application No. filed on and as amended under PCT Article 19 on (if any).

ACKNOWLEDGEMENT OF REVIEW OF PAPERS AND DUTY OF CANDOR

I hereby state that I have reviewed and understand the contents of the above identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information which is material to the examination of this application in accordance with Title 37 CFR 1.56(a).

PRIORITY CLAIM

I hereby claim foreign priority benefits under Title 35, United States Code, Section 119 of any foreign application(s) for patent or inventors certificate or of any PCT International application(s) designating at least one country other than the United States of America listed below and have also identified below any foreign application(s) for patent or inventor's certificate of any PCT International application(s) designating at least one country other than the United States of America filed by me on the same subject matter having a filing date before that of the application(s) of which priority is claimed.

(d) ☒ No such applications have been filed.
(e) ☐ Such applications have been filed as follows.

NOTE: Where item (c) is entered above and the International Application which designated the U.S. claimed priority, check item (e), enter the details below and make the priority claim.

Earliest Foreign Application(s), if any, filed within 12 months (6 months for Design) prior to this U.S. Application

Country	Application No.	Date of Filing (Day, Month, Year)	Priority Claimed 35 USC 119

All Foreign Application(s), if any, Filed More Than 12 Months (6 Months for Design) Prior to This U.S. Application)

Docket No 33343-01
Patent

CLAIM FOR BENEFIT OF PRIOR U.S. PROVISIONAL APPLICATION(S)
(35 U.S.C. § 119(E))

I hereby claim the benefit under Title 35, United States Code, § 119(e) of any United States provisional application(s) listed below:

PROVISIONAL APPLICATION NUMBER	FILING DATE
60/052071	July 9, 1997

POWER OF ATTORNEY

As a named inventor, I hereby appoint the following attorney(s) and/or agent(s) to prosecute this application and transact all business in the Patent and Trademark Office connected therewith.

William H. Calnan	29,520
Charles F. Costello, Jr.	27,324
Gregory M. Hill	31,369
John W. Hogan, Jr	32,703
Joseph M. Mazzarese	32,803
Barbara L. Renda	27,626

☒ Attached as part of this declaration and power of attorney is the authorization of the above-named attorney(s) to accept and follow instructions from my representative(s).

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Tel. No. (973) 683-2153

Docket No. 33343-01
Patent

CLAIM FOR BENEFIT OF EARLIER U.S./PCT APPLICATION(S) UNDER 35 U.S.C. 120

I hereby claim the benefit under Title 35, United States Code, Section 120 of any United States application(s) designating the United States of America that is/are listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in that/those prior application(s) in the manner provided by the first paragraph of Title 35, United States Code, Section 112. I acknowledge the duty to disclose material information as defined in Title 37, Code of Federal Regulations, Section 1.56(a) which occurred between the filing date of the prior application(s) and the national or PCT International filing date of this application.

PRIOR U.S. APPLICATIONS OR PCT INTERNATIONAL APPLICATIONS DESIGNATING THE U.S. FOR BENEFIT UNDER 35 USC 120

U.S. Applications		**Status (Check One)**		
U.S. Applications	U.S. Filing Date	Patented	Pending	Abandoned
1.		☐	☐	☐
2.		☐	☐	☐

PCT Applications Designating U.S.		
PCT APPLICATION NO.	PCT FILING DATE	U.S. SERIAL NO. ASSIGNED (if any)
3.		
4.		

Docket No. 33343-01
Patent

DECLARATION

I hereby declare that all statements herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements are made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

SIGNATURE(S)

Full name of SOLE OR FIRST INVENTOR **Guanglin Sun**

Inventor's Signature _____ Date 6/9/98
Country of Citizenship **P.R.C.**
Residence **New Jersey**
Post Office Address **80 Marion Drive, Plainsboro, New Jersey**

Full name of SECOND JOINT INVENTOR **Fakhruddin Ahmed**

Inventor's Signature _____ Date 06/09/98
Country of Citizenship **USA**
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Full name of THIRD JOINT INVENTOR **Bruce Christian Black**

Inventor's Signature _____ Date _____
Country of Citizenship **USA**
Residence **Pennsylvania**
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Full name of FOURTH JOINT INVENTOR

Inventor's Signature _____ Date _____
Country of Citizenship
Residence
Post Office Address

Docket No. 33343-01
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(Original, Design, Supplemental, Divisional, Continuation,CIP)

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Earliest Foreign Application(s), if any, filed within 12 months (6 months for Design) prior to this U.S. Application

Country	Application No.	Date of Filing (Day, Month, Year)	Priority Claimed 35 USC 119

All Foreign Application(s), if any, Filed More Than 12 Months (6 Months for Design) Prior to This U.S. Application)

Docket No. 33343-01
Patent

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Docket No. 33343-01
Patent

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PRIOR U.S. APPLICATIONS OR PCT INTERNATIONAL APPLICATIONS DESIGNATING THE U.S. FOR BENEFIT UNDER 35 USC 120

U.S. Applications		**Status (Check One)**		
U.S. Applications	U.S. Filing Date	Patented	Pending	Abandoned
1.		☐	☐	☐
2.		☐	☐	☐

PCT Applications Designating U.S.		
PCT APPLICATION NO.	PCT FILING DATE	U.S. SERIAL NO. ASSIGNED (if any)
3.		
4.		

Docket No. 33343-01
Patent

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SIGNATURE(S)

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Inventor's Signature_____ Date_____
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Post Office Address **80 Marion Drive, Plainsboro, New Jersey**

Full name of SECOND JOINT INVENTOR **Fakhruddin Ahmed**

Inventor's Signature_____ Date_____
Country of Citizenship **USA**
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Full name of THIRD JOINT INVENTOR **Bruce Christian Black**

Inventor's Signature Bruce Christian Black Date June 9, 1998
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Full name of FOURTH JOINT INVENTOR

Inventor's Signature_____ Date_____
Country of Citizenship
Residence
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